6.3.3 Erlenmeyer Flasks. 125-ml, 24/40 standard taper.

6.3.4 Membrane Filters. Millipore SCWPO 4700, or equivalent.

6.3.5 Filtration Apparatus. Millipore vacuum filtration unit, or equivalent, for use with the above membrane filter.

6.3.6 Volumetric Flasks. 100-ml, 250-ml, and 1000-ml.

7.0 Reagents and Standards

NOTE: Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available; otherwise, use the best available grade.

7.1 Sample Collection. The following reagents are needed for sample collection:

7.1.1 Filter. Gelman Spectro Grade, Reeve Angel 934 AH, MSA 1106 BH, all with lot assay for Pb, or other high-purity glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency (<0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. Conduct the filter efficiency test using ASTM D 2986–71, 78, or 95a (incorporated by reference—see §60.17) or use test data from the supplier's quality control program.

7.1.2 Silica Gel, Crushed Ice, and Stopcock Grease. Same as Method 5, Sections 7.1.2, 7.1.4, and 7.1.5, respectively.

7.1.3 Water. Deionized distilled, to conform to ASTM D 1193–77 or 91, Type 3 (incorporated by reference—see §60.17). If high concentrations of organic matter are not expected to be present, the potassium permanganate test for oxidizable organic matter may be omitted.

7.1.4 Nitric Acid, 0.1 N. Dilute 6.5 ml of concentrated HNO_3 to 1 liter with water. (It may be desirable to run blanks before field use to eliminate a high blank on test samples.)

7.2 Sample Recovery. 0.1 N HNO_3 (Same as in Section 7.1.4 above).

7.3 Sample Analysis. The following reagents and standards are needed for sample analysis:

7.3.1 Water. Same as in Section 7.1.3.

7.3.2 Nitric Acid, Concentrated.

7.3.3 Nitric Acid, 50 Percent (v/v). Dilute 500 ml of concentrated HNO_3 to 1 liter with water.

7.3.4 Stock Lead Standard Solution, 1000 μg Pb/ml. Dissolve 0.1598 g of lead nitrate [$Pb(NO_3)_2$] in about 60 ml water, add 2 ml concentrated HNO_3, and dilute to 100 ml with water.

7.3.5 Working Lead Standards. Pipet 0.0, 1.0, 2.0, 3.0, 4.0, and 5.0 ml of the stock lead standard solution (Section 7.3.4) into 250-ml volumetric flasks. Add 5 ml of concentrated HNO_3 to each flask, and dilute to volume with water. These working standards contain 0.0, 4.0, 8.0, 12.0, 16.0, and 20.0 μg Pb/ml, respectively. Prepare, as needed, additional standards at other concentrations in a similar manner.

7.3.6 Air. Suitable quality for atomic absorption spectrophotometry.

7.3.7 Acetylene. Suitable quality for atomic absorption spectrophotometry.

7.3.8 Hydrogen Peroxide, 3 Percent (v/v). Dilute 10 ml of 30 percent H_2O_2 to 100 ml with water.

8.0 Sample Collection, Preservation, Storage, and Transport

8.1 Pretest Preparation. Follow the same general procedure given in Method 5, Section 8.1, except that the filter need not be weighed.

8.2 Preliminary Determinations. Follow the same general procedure given in Method 5, Section 8.2.

8.3 Preparation of Sampling Train. Follow the same general procedure given in Method 5, Section 8.3, except place 100 ml of 0.1 N HNO_3 (instead of water) in each of the first two impingers. As in Method 5, leave the third impinger empty and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger. Set up the train as shown in Figure 12–1.

8.4 Leak-Check Procedures. Same as Method 5, Section 8.4.

8.5 Sampling Train Operation. Same as Method 5, Section 8.5.

8.6 Calculation of Percent Isokinetic. Same as Method 5, Section 8.6.

8.7 Sample Recovery. Same as Method 5, Sections 8.7.1 through 8.7.6.1, with the addition of the following:

8.7.1 Container No. 2 (Probe).

8.7.1.1 Taking care that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover sample matter and any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with 0.1 N HNO_3 and placing the wash into a glass sample storage container. Measure and record (to the nearest 2 ml) the total amount of 0.1 N HNO_3 used for these rinses. Perform the 0.1 N HNO_3 rinses as follows:

8.7.1.2 Carefully remove the probe nozzle, and rinse the inside surfaces with 0.1 N HNO_3 from a wash bottle while brushing with a stainless steel, Nylon-bristle brush. Brush until the 0.1 N HNO_3 rinse shows no visible particles, then make a final rinse of the inside surface with 0.1 N HNO_3.

8.7.1.3 Brush and rinse with 0.1 N HNO_3 the inside parts of the Swagelok fitting in a similar way until no visible particles remain.

8.7.1.4 Rinse the probe liner with 0.1 N HNO_3. While rotating the probe so that all inside surfaces will be rinsed with 0.1 N

HNO_3, tilt the probe, and squirt 0.1 N HNO_3 into its upper end. Let the 0.1 N HNO_3 drain from the lower end into the sample container. A glass funnel may be used to aid in transferring liquid washes to the container. Follow the rinse with a probe brush. Hold the probe in an inclined position, squirt 0.1 N HNO_3 into the upper end of the probe as the probe brush is being pushed with a twisting action through the probe; hold the sample container underneath the lower end of the probe, and catch any 0.1 N HNO_3 and sample matter that is brushed from the probe. Run the brush through the probe three times or more until no visible sample matter is carried out with the 0.1 N HNO_3 and none remains on the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times, since metal probes have small crevices in which sample matter can be entrapped. Rinse the brush with 0.1 N HNO_3, and quantitatively collect these washings in the sample container. After the brushing, make a final rinse of the probe as described above.

8.7.1.5 It is recommended that two people clean the probe to minimize loss of sample. Between sampling runs, keep brushes clean and protected from contamination.

8.7.1.6 After ensuring that all joints are wiped clean of silicone grease, brush and rinse with 0.1 N HNO_3 the inside of the from half of the filter holder. Brush and rinse each surface three times or more, if needed, to remove visible sample matter. Make a final rinse of the brush and filter holder. After all 0.1 N HNO_3 washings and sample matter are collected in the sample container, tighten the lid on the sample container so that the fluid will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether leakage occurs during transport. Label the container to identify its contents clearly.

8.7.2 Container No. 3 (Silica Gel). Note the color of the indicating silica gel to determine if it has been completely spent, and make a notation of its condition. Transfer the silica gel from the fourth impinger to the original container, and seal. A funnel may be used to pour the silica gel from the impinger and a rubber policeman may be used to remove the silica gel from the impinger. It is not necessary to remove the small amount of particles that may adhere to the walls and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for Container No. 3 in Section 11.4.2.

8.7.3 Container No. 4 (Impingers). Due to the large quantity of liquid involved, the impinger solutions may be placed in several containers. Clean each of the first three impingers and connecting glassware in the following manner:

8.7.3.1. Wipe the impinger ball joints free of silicone grease, and cap the joints.

8.7.3.2. Rotate and agitate each impinger, so that the impinger contents might serve as a rinse solution.

8.7.3.3. Transfer the contents of the impingers to a 500-ml graduated cylinder. Remove the outlet ball joint cap, and drain the contents through this opening. Do not separate the impinger parts (inner and outer tubes) while transferring their contents to the cylinder. Measure the liquid volume to within 2 ml. Alternatively, determine the weight of the liquid to within 0.5 g. Record in the log the volume or weight of the liquid present, along with a notation of any color or film observed in the impinger catch. The liquid volume or weight is needed, along with the silica gel data, to calculate the stack gas moisture content (see Method 5, Figure 5–6).

8.7.3.4. Transfer the contents to Container No. 4.

NOTE: In Sections 8.7.3.5 and 8.7.3.6, measure and record the total amount of 0.1 N HNO_3 used for rinsing.

8.7.3.5. Pour approximately 30 ml of 0.1 N HNO_3 into each of the first three impingers and agitate the impingers. Drain the 0.1 N HNO_3 through the outlet arm of each impinger into Container No. 4. Repeat this operation a second time; inspect the impingers for any abnormal conditions.

8.7.3.6. Wipe the ball joints of the glassware connecting the impingers free of silicone grease and rinse each piece of glassware twice with 0.1 N HNO_3; transfer this rinse into Container No. 4. Do not rinse or brush the glass-fritted filter support. Mark the height of the fluid level to determine whether leakage occurs during transport. Label the container to identify its contents clearly.

8.8 Blanks.

8.8.1 Nitric Acid. Save 200 ml of the 0.1 N HNO_3 used for sampling and cleanup as a blank. Take the solution directly from the bottle being used and place into a glass sample container labeled ''0.1 N HNO_3 blank.''

8.8.2 Filter. Save two filters from each lot of filters used in sampling. Place these filters in a container labeled ''filter blank.''

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures.